Polymer Science U.S.S.R. Vol. 21, pp. 2985–2990.
0032-3950/79/1201-2985$07.50/0

CRYSTALLIZATION OF OLIGO(ETHYLENE GLYCOL) IN THE PRESENCE OF CARBON TETRACHLORIDE*

N. K. Dvoyashkin, A. I. Maklakov and V. S. Smirnov

V. I. Ul'yanov-Lenin State University, Kazan'

(*Received* 5 *October* 1978)

The kinetics of crystallization of oligo(ethylene glycol) in the presence of carbon tetrachloride, have been studied by the pulsed NMR method. It was found that the overall rate of crystallization and the shape of the isotherms are determined by the initial concentration of the solvent in the system, and by the change in composition of the amorphous phase during the course of the process. It is shown that the results can be explained qualitatively by the theory of crystallization of polymers in the presence of low molecular liquids.

The kinetics of crystallization from melts have been studied fairly well at the present time, but in spite of this there are very few papers devoted to study of the effect of low molecular liquids on this phase stage. Meanwhile there are some special features of crystallization of polymers in the presence of solvents [1, 2].

In the present paper we report a study of the effect of carbon tetrachloride on the kinetics of crystallization of oligo(ethylene glycol) (OEG) by the pulsed NMR method.

The OEG had a number average molecular weight of 2000, as determined by end group analysis, and the CCl_4 was the pure-for-analysis grade. The volume fractions of oligomer, φ_2, in the specimens used for measurement were 1·00 (1); 0·926 (2); 0·746 (3); 0·56 (4); 0·44 (5); 0·36 (6); 0·30 (7) and 0·25 (8). The numbers in brackets will be used subsequently to denote the particular specimens. The mixtures of given composition were prepared in glass tubes, which were then sealed and held at 70° for 10–12 hr [3] and then kept at room temperature. Immediately before the measurements the specimens were melted at 70° and held at that temperature for 2 hr.

Isothermal crystallization was carried out directly in the measuring cell of the NMR relaxometer [4], by the method described in reference [5], at temperatures in the range of 7·5–40°C. The crystallization temperature, T_c, was controlled within $\pm 0{\cdot}2°$. The instantaneous degree of crystallization [5, 6] and the proton spin-spin relaxation time T_2 [7], were measured during the course of crystallization. The melting points, T_m, were determined in a laboratory DTA apparatus, from the endothermal melting peak [8].

The relaxation of the cross-sectional magnetization of crystallizable specimens can be represented by the equation

$$A(t)=A_{0a}\exp\left(-\frac{t}{T_{2a}}\right)+A_{0c}f(t),$$

* Vysokomol. soyed. **A21**: No. 12, 2702–2707, 1979.